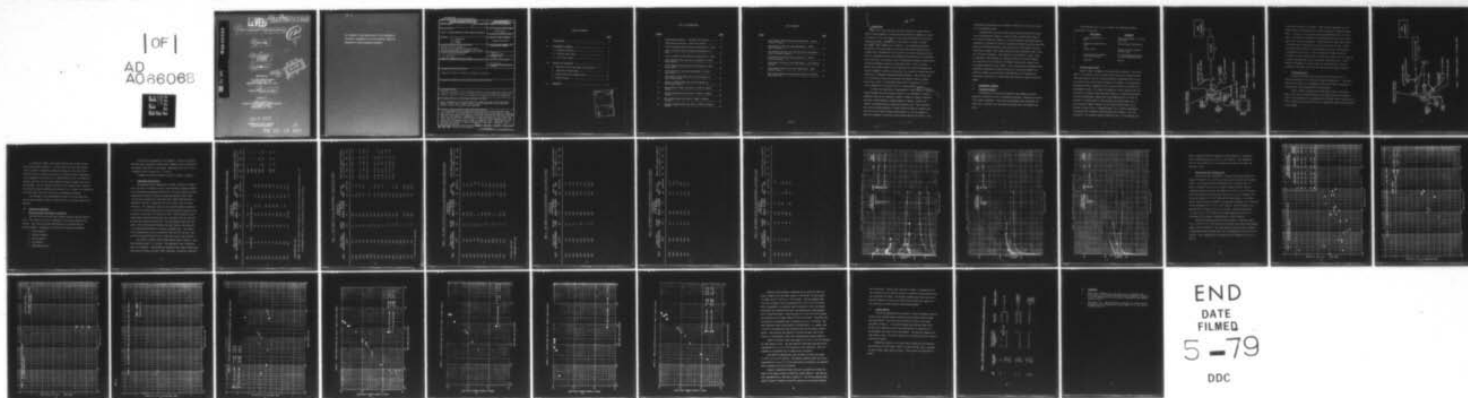


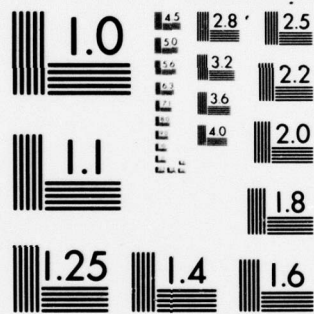
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9 STUDY OF LASER EFFECTS ON HEAT SHIELD MATERIALS

9 FINAL REPORT

BY

10 P.D./ZAVITSANOS,
J.A./GOLDEN
W.G./BROWNE

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RE-ENTRY AND ENVIRONMENTAL SYSTEMS DIVISION
3198 CHESTNUT STREET
PHILADELPHIA, PA 19101

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) An experimental study of CO ₂ laser heating of heat shield materials has been conducted. The interaction of carbon phenolic, silica phenolic, phenolic asbestos, ATJ graphite and phenolic resin with a CW 100 watt and a CW 10 KW CO ₂ laser has been examined. Experiments at power densities of 10 ¹ -10 ⁴ watts/cm ² induce mass losses of 1-4x10 ⁻⁵ g/joule in phenolic-containing materials, mass losses from ATJ graphite are an order of magnitude lower. Sampling and mass spectrometric analysis of gases emanating from the laser interaction zone was also carried out.		

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I. INTRODUCTION

Materials of the type used for heat shields on re-entry vehicles undergo varying degrees of property and characteristic changes as a result of exposure to laser radiation in a space type environment at flux levels ranging from about 1 watt/cm² up to flux levels in excess of 10,000 watts/cm². These changes are, in general: (a) outgassing and depolymerization of the resin binder at low heat fluxes with potential loss in strength and/or delamination of the shield; (b) surface charring and high rates of outgassing and material decomposition/vaporization at intermediate heat fluxes and; (c) excessive rates of outgassing and material vaporization and shield thickness reduction at high heat fluxes. Thus a re-entry vehicle heat shield exposed to laser radiation in a space environment may provide inadequate protection during entry for reasons of reduced structural capability or inadequate remaining shield thickness. Also the heat soak into the shield and structure resulting from exposure times of several minutes may result in unacceptably high levels of temperature for the vehicle substructure or payload prior to or during entry.

To assess the importance of exoatmospheric laser heating to weapon system missions involving re-entry vehicles, it is necessary to conduct a systematic study of the response of generic classes of thermal protection materials (carbon phenolic, asbestos phenolic, phenolic refrasil and carbon) to varying radiative fluxes and exposure times. With these data and a detailed understanding of thermal protection material behavior and specific re-entry vehicle mission requirements, it will be possible to assess the adequacy of existing weapon systems against this threat. Also,

countermeasure approaches can be planned or defined if the threat is found to be unacceptably severe.

Although laser radiation exposure above about 500 watts/cm² results in very rapid material removal or loss the laser radiation flux range of 10 to about 100 watts/cm² appears to be of the greatest importance from the standpoint of studying the radiative response of thermal protection materials. Under these heating conditions, particularly with fluctuating or cyclical temperatures, organic resin bonded refractory fiber composites tend to have poor structural integrity. The tendency of the composite to warp, crack or delaminate under exposure is a complex function of the char forming characteristics of the resin and the thermal conductivity, surface area and chemical stability of the fibers. Thus the selection of phenolic based composites reinforced with carbon, refrasil or silica and asbestos for study will result in much detailed understanding of the importance of fiber-matrix interaction in controlling laser radiation induced structural damage.

II. EXPERIMENTAL APPROACH

A. Parameters Measured

A system was set up to heat cylindrical plug samples of shield materials in vacuum or in air at atmospheric pressure. Samples were heated by CO₂ laser irradiation. Two series of experiments were performed, the first with a 100 watt C.W. laser and the second with a 10 kilowatt C.W. laser.

Instrumentation was set up to measure the following parameters during sample irradiation:

	<u>MEASUREMENT</u>	<u>INSTRUMENT</u>
1	Surface Temperature vs. Time	Radiation Pyrometer (2-2.5 μ m pass band)
2	Subsurface Temperature vs. Time	Chromel-Alumel Thermocouple
3	Pressure Rise	Wallace & Tiernan Absolute Pressure Gage
4	Gaseous Species Produced from Laser Heating	Gas Chromatograph and Time of Flight Mass Spectrometer
5	Mass Loss	Balance

B. 100 Watt Laser System

Figure 1 shows a diagram of the experimental apparatus. The output beam (1.5 cm diameter) from a 100 watt CW CO₂ laser (A) is passed through a KBr beam splitter (B) and a Germanium meniscus condensing lens (C) onto the surface of the shield material sample to be heated. A chromel-alumel thermocouple bead is imbedded on axis in the cylindrical plug sample midway between the ends. The sample is mounted in a pyrex vacuum vessel (D) connected to a sampling flask (E) which collects an integrated gas sample for gas chromatographic analysis and a gas sampling valve (F) which is attached to a Bendix Model 12 Time-of-Flight mass spectrometer (G). A mass spectrum of gases produced from the heated shield sample can be recorded at about 10 sec. intervals during a run. Surface temperature is measured with an IRCON Co model 300 Radiation Pyrometer (2-2.5 μ m band pass). The pyrometer detects radiation from a 1.5 mm diameter spot

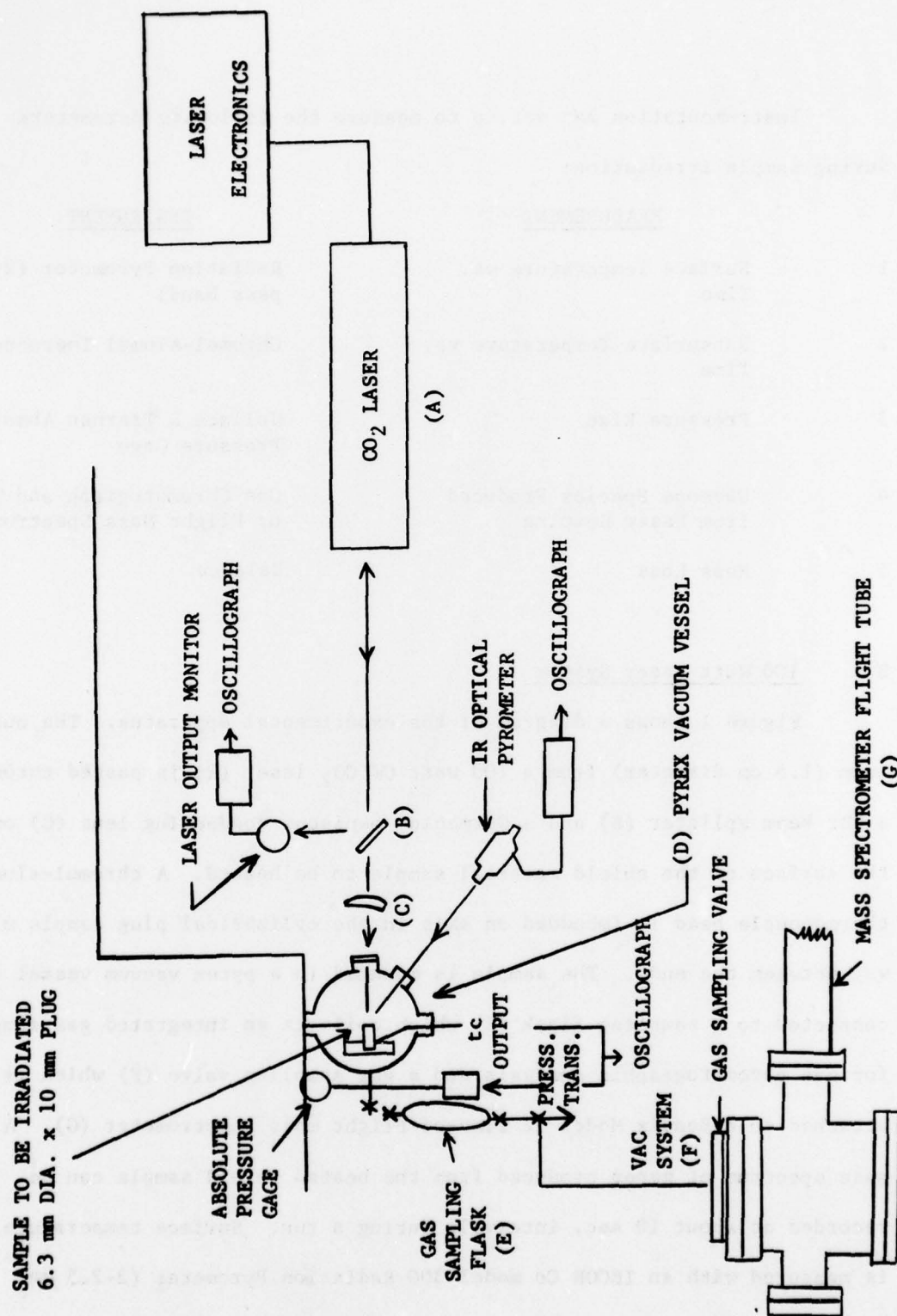


FIGURE 1 . EXPERIMENTAL APPARATUS - 100 WATT LASER SYSTEM

on the front surface of the sample. Power density (watts/cm²) at the sample surface is monitored with a Coherent Radiation Co Model 201 Power Meter. A portion of the CO₂ laser beam (approximately 10% of the total power in the beam) is reflected from the KBr beam splitter into the laser power meter for this measurement. Calibration of the laser power monitor is accomplished by replacing the pyrex vacuum vessel (D) with a second laser power meter located in the same plane as the plug sample surface. A measurement of the ratio of power transmitted through all optical components to that reflected into the laser power monitor (E) as a function of laser operating power level provides the calibration data. A multi-channel oscillograph is used to record the time history of pressure, temperature, and power level from the instrumentation shown in Figure 1.

C. 10 KW Laser System

The GE 10 KW CW CO₂ Laser Facility was used for this work. A detailed description of the facility is given in Reference 1. The output beam from this laser is 9 cm diameter and the power level in the beam is variable from a few hundred watts to ten kilowatts.

Figure 2 shows a diagram of the experimental setup. The output beam of the laser (A) passes through a salt beam splitter (B) to monitor power at the detector (C). The main beam is reflected from a flat mirror (D) onto a concave mirror (E) which serves to condense the beam onto the surface of the sample.

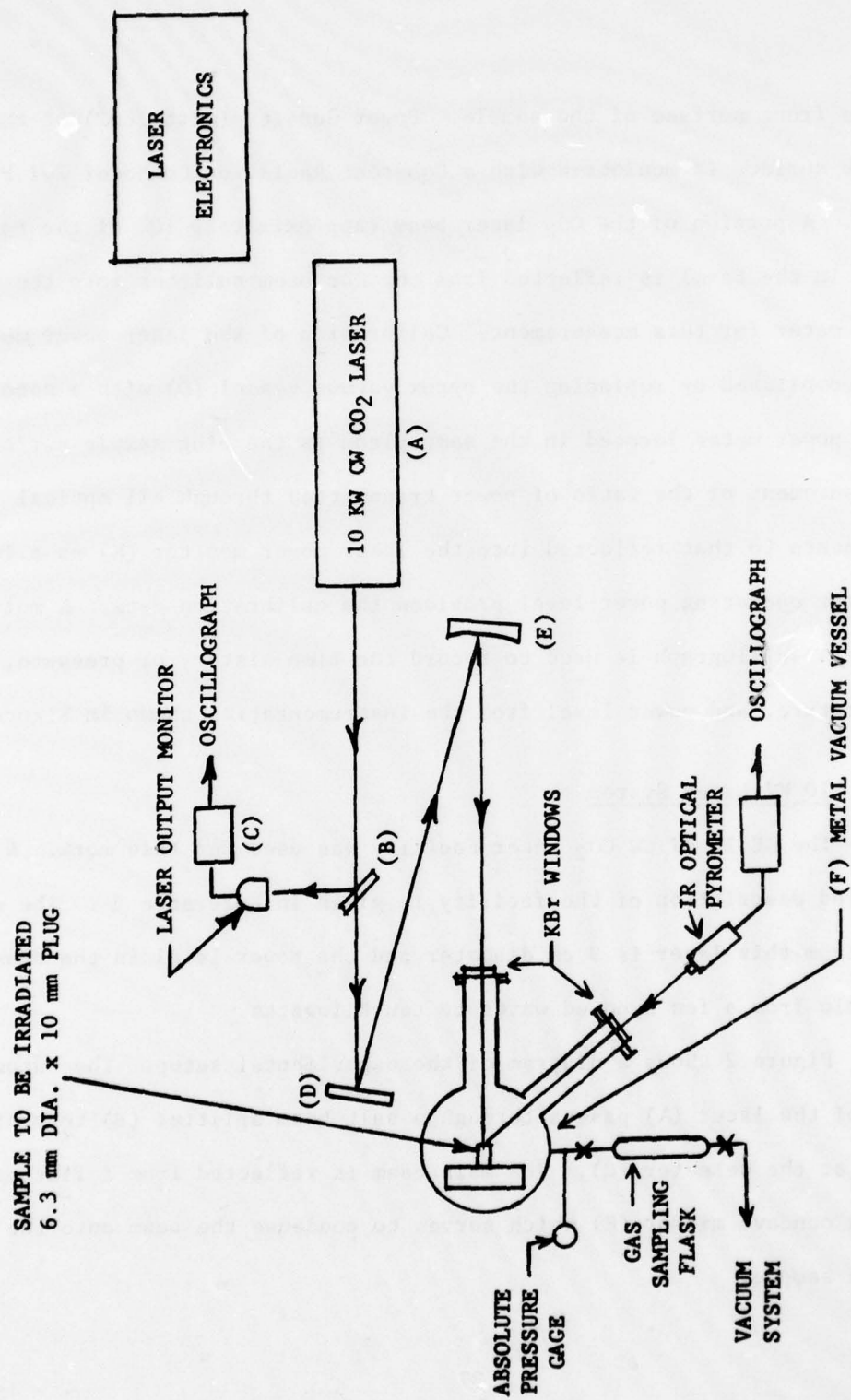


FIGURE 2 . EXPERIMENTAL APPARATUS - 10 KW LASER SYSTEM

A cylindrical copper vacuum vessel (F) was used in place of the pyrex vessel shown in Figure 1. In this vessel the laser beam entrance and the pyrometer viewing KBr windows were mounted at the end of 30 cm extension tubes to minimize the build-up of ablation product condensation. This arrangement was effective up to 2000 W/cm^2 heat load. At 2000 W/cm^2 graphite vaporizes from the shield sample and forms an opaque film on the salt windows. The film absorbs a portion of the incoming laser radiation, the window is rapidly overheated and fractures. As a result, all experiments above this power level were carried out at atmospheric pressure.

The remainder of the instrumentation used in the high power work was the same as shown in Figure 1 except that no in situ mass spectra were obtained.

III. RESULTS AND DISCUSSION

A. Materials Studied and Range of Conditions

Three R/V shield materials were studied along with GE 9ILD phenolic resin. This resin is one of the components of the carbon phenolic heat shields. Also several baseline experiments were performed with ATJ graphite samples. Following is a listing of the materials examined:

1. Carbon Phenolic
2. Phenolic Asbestos
3. Silica Phenolic
4. ATJ Graphite
5. 9ILD Phenolic Resin

Cylindrical plug samples (6.3 mm diameter x 10 mm) of the above materials were irradiated at power levels ranging from 10 to 9100 W/cm² at exposure times from 1 to 60 seconds. Experiments were run in air at 1 atmosphere and in vacuum (.05 - 1.5 torr).

A summary of the data obtained is given in Tables 1 through 6.

B. Temperature History Data

The measured surface temperature is shown in Figures 3 through 5 versus the laser irradiation time for carbon phenolic, phenolic asbestos and silica phenolic specimens respectively. The carbon phenolic surface temperatures in Figure 3 display a pronounced maxima at early times < 4 secs when irradiated by 10 KW laser heat fluxes ≥ 650 watts/cm². A substantial falloff in temperature occurs at longer times for these conditions. The temperature peak and rapid falloff behavior exhibited at 10 KW laser fluxes ≥ 650 watts/cm² is associated with a change in the emissivity of the char as a function of time. Carbon phenolic surface temperatures increase monotonically with irradiation time for 100 W CO₂ CW laser heat fluxes ≤ 44 watts/cm². The intermediate laser heat flux range ~ 100 to 500 watts/cm² displays the same temporal characteristic of a broad maxima followed by a virtual isothermal zone. The anomaly of the juxtaposition of the 115 watts/cm² data with the 100 watt laser and the 325 watts/cm² data with the 10 KW laser has not been resolved.

The phenolic asbestos surface temperatures shown in Figure 4 reach their maximum values in ~ 5 seconds. The remainder of the irradiation period is uneventful. Carbon phenolic specimens reach higher temperatures than phenolic asbestos specimens under comparable irradiation conditions.

TABLE 1. DATA SUMMARY 100 WATT CO₂ LASER EXPERIMENTS---CARBON PHENOLIC SAMPLES

RUN #	POWER DENSITY (AT SAMPLE SURFACE) w/cm ²	EXPOSURE TIME sec	TEMPERATURE AT END OF RUN		MID POINT OF SAMPLE °C	MASS LOSS TOTAL mg	MASS LOSS RATE mg/cm ² -sec	MAJOR GASEOUS ABLATION PRODUCTS ppm				
			SURFACE °C	°C				H ₂	H ₂ O	CO	CO ₂	HC's
LE1	10	60	260	50	*			8600	--	--	--	
LE2	GC BACKGROUND CALIBRATION											
LE3	10	60	412	220		5.5	.290	12800	--	--	*	
LE4	15	60	607	295		16.3	.860	11000	--	114	*	
LE5	23	60	740	*		15.0	.791	4878	184	744	100	
LE15	36	60	790	180		12.5	.656					
LE6	44	60	820	246		18.0	.949	4800	27	512	50	
LE7	44	60	882	239		15.5	.817	2400	54	92	75	
LE8	100	60	1050	280		31.5	1.66					
LE9	115	60	1390	*		29.0	1.53					
LE16	135	60	1335	270		29.0	1.53					
LE43	4840	4.1	NO DATA			4.8	11.70					

* MEASUREMENT NOT MADE

NOTES: THE LASER BEAM DIAMETER OF THE SAMPLE WAS .635 CM FOR ALL RUNS EXCEPT LE43 WHICH WAS .1 CM

ALL EXPERIMENTS WERE RUN AT .05 TORR EXCEPT LE43 WHICH WAS AT 760 TORR.

TABLE 2. DATA SUMMARY 10 KW CO₂ LASER EXPERIMENTS---CARBON PHENOLIC SAMPLES

RUN #	POWER DENSITY (AT SAMPLE SURFACE) w/cm ²	EXPOSURE TIME sec	TEMPERATURE AT END OF RUN		MASS LOSS TOTAL mg	MASS LOSS RATE mg/cm ² -sec	MAJOR GASEOUS ABLATION PRODUCTS ppm				
			SURFACE °C	MID POINT OF SAMPLE °C			H ₂	H ₂ O	CO	CO ₂	HC's
LE31	325	30	1280		62	6.5	23.27	56.61	10.86	5.62	3.64
*LE17	340	60	1570		90.5	4.8					
LE18	630	15	2180		83.5	17.7	19.92	34.9	3.06	1.22	9.79
LE25	650	15	1440		73.7	15.5	25.91	4.84	10.94	4.42	4.09
LE40A	650	15	1250		72.5		28.33	48.36	12.44	4.42	3.52
LE40B	650	11.5	1280								
LE20	655	15	2635		90.0	18.9	11.98	23.67	31.77	1.13	
LE28	970	10	1675		79.4	25.1	19.85	17.06	8.53	1.05	3.54
LE19	1000	15	2325		100.0	21.1	21.35	26.5	3.81	1.35	13.87
LE34	1250	8	1600		80.6		27.53	49.07	11.67	3.71	4.55
LE37	1500	6.5	1825		76.0		31.51	47.27	12.98	3.61	4.26
*LE21	2000	7.5	2920		156.0	65.7	15.89	6.35	19.95	2.37	
LE22	4050	3.75	2800		159.0	133.9					
*LE23	5000	3			132.5						
LE48	5000	2	2150		80.3	126.8					
LE51	7150	1.4	1890		83.5	188.4					
LE54	9100	1.1	1810		75.3	216.4					

*IR WINDOW CRACKED ON THESE RUNS

TABLE 3. DATA SUMMARY 100 WATT AND 10 KW CO₂ LASER EXPERIMENTS---PHENOLIC ASBESTOS SAMPLES

RUN #	POWER DENSITY (AT SAMPLE SURFACE) w/cm ²	EXPOSURE TIME sec	TEMPERATURE AT END OF RUN		MASS LOSS TOTAL mg	MASS LOSS RATE mg/cm ² -sec	MAJOR GASEOUS ABLATION PRODUCTS			
			SURFACE °C	MID POINT OF SAMPLE °C			ppm			
							H ₂	H ₂ O	CO	CO ₂
LE32	350	30	1280		111.3	11.7				
LE26	600	15	1675		118.9	25.1				
LE29	970	10	1750		123.5	39.1				
LE44	50*	60	X		37.1	1.95				
LE45	100*	33	X		35.8	3.43				
LE35	1250	8	1850		130.4	51.5				
LE38	1500	6.5	1550		130.9	63.6				
LE47	5000	2	1700		101.9	160.9				
LE42	5095*	4.5	X		9.7	274.7				
LE41	5350*	4.1	X		9.4	292.1				
LE50	7150	1.4	2050		105.5	238.1				
LE53	9100	1.1	1950		107.8	309.8				

*100 WATT LASER RUNS

X = MEASUREMENT NOT MADE

TABLE 4. DATA SUMMARY 10 KW CO₂ LASER EXPERIMENTS----SILICA PHENOLIC SAMPLES

RUN #	POWER DENSITY (AT SAMPLE SURFACE) w/cm ²	EXPOSURE TIME sec	TEMPERATURE		MASS LOSS TOTAL mg	MASS LOSS RATE mg/cm ² -sec	MAJOR GASEOUS ABLATION PRODUCTS					
			AT END OF RUN				ppm					
			SURFACE °C	MID POINT OF SAMPLE °C			H ₂	H ₂ O	CO	CO ₂	HC's	
LE33	325	30	1210		49.6	5.2						
LE27	620	15	1650		60.6	12.8						
LE30	1030	10	1350		100.9	31.9						
LE36	1250	8	1675		90.2	35.6						
LE39	1500	6.5	1800		93.2	45.3						
LE46	5000	2	1825		74.3	117.3						
LE49	7150	1.4	1850		113.1	255.0						
LE52	9100	1.1	1810		92.8	266.0						

TABLE 5. DATA SUMMARY 10 KW CO₂ LASER EXPERIMENTS---ATJ GRAPHITE SAMPLES

RUN #	POWER DENSITY (AT SAMPLE SURFACE) w/cm ²	EXPOSURE TIME sec	TEMPERATURE		MASS LOSS TOTAL mg	mg/cm ² -sec	MAJOR GASEOUS ABLATION PRODUCTS				
			SURFACE °C	MID POINT OF SAMPLE °C			ppm				
							H ₂	H ₂ O	CO	CO ₂	HC's
LE48A	5000	2.0	1350		0.2	0.31					
LE51A	7150	1.4	1700		2.3	5.20					
LE54A	9100	1.1	2100		2.5	7.20					
LE56	9100	3.0	2550		15.7	16.6					
LE55	9100	5.0	2400		19.7	12.5					
LE57	9100	8.0	2400		44.8	17.7					

TABLE 6. DATA SUMMARY 100 WATT CO₂ LASER EXPERIMENTS---91LD PHENOLIC RESIN

RUN #	POWER DENSITY (AT SAMPLE SURFACE) w/cm^2	EXPOSURE TIME sec	TEMPERATURE		MASS LOSS TOTAL mg	MASS LOSS RATE mg/cm^2 -sec	MAJOR GASEOUS ABLATION PRODUCTS					
			AT END OF RUN				ppm					
			SURFACE °C	MID POINT OF SAMPLE °C			H ₂	H ₂ O	CO	CO ₂	HC's	
LE10	10	60	520	75	10	.527						
LE11	87	60	1300	150	--	----						
LE12	23	60	900	120	35	1.84						
LE13	102	60	1645	---	78.5	4.14						
LE14	73	60	1305	180	58	3.05						

FIGURE 3. LASER IRRADIATED CARBON PHENOLIC TEMPERATURE VS. TIME

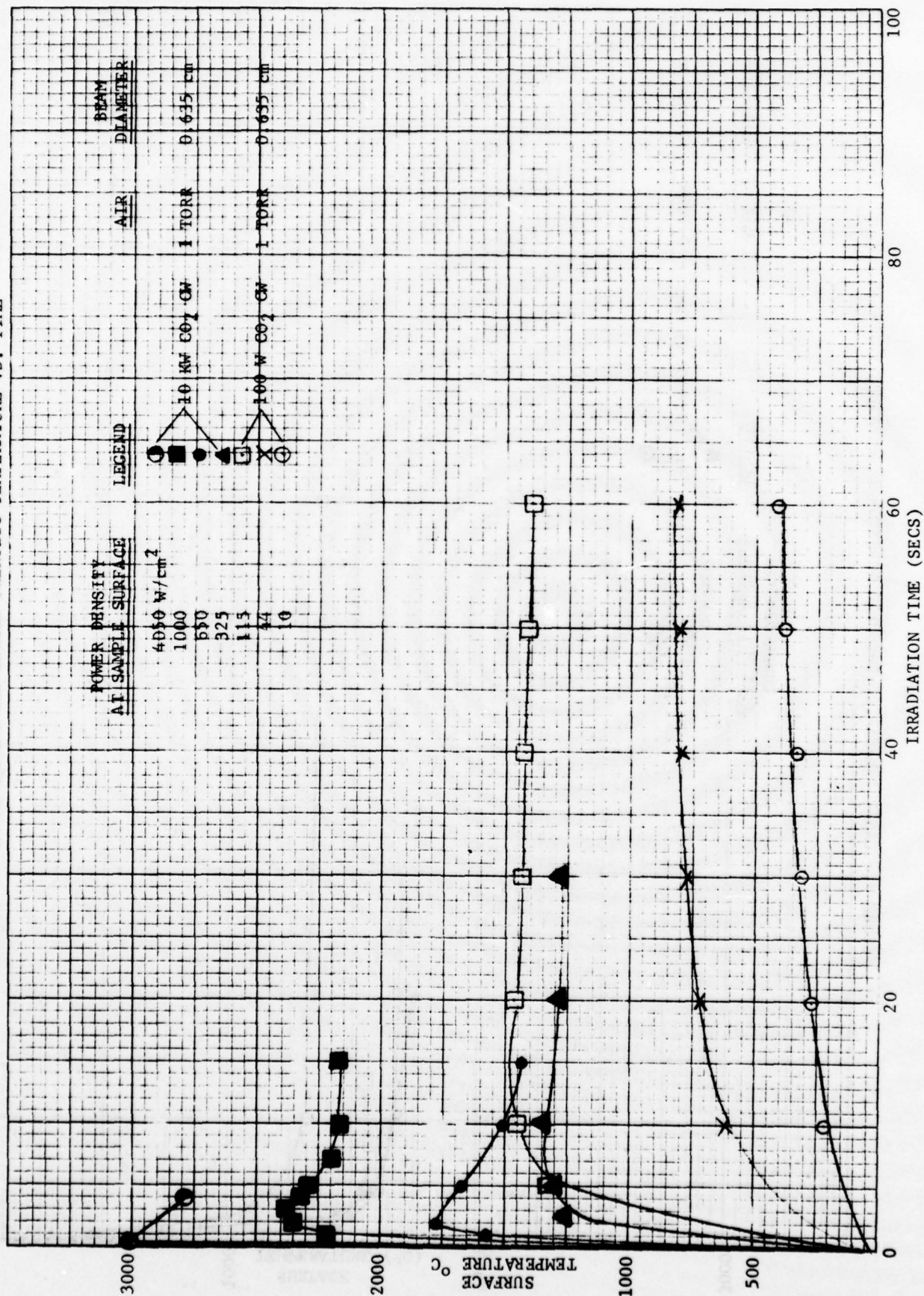


FIGURE 4. LASER IRRADIATED PHENOLIC ASBESTOS TEMPERATURE VS. TIME

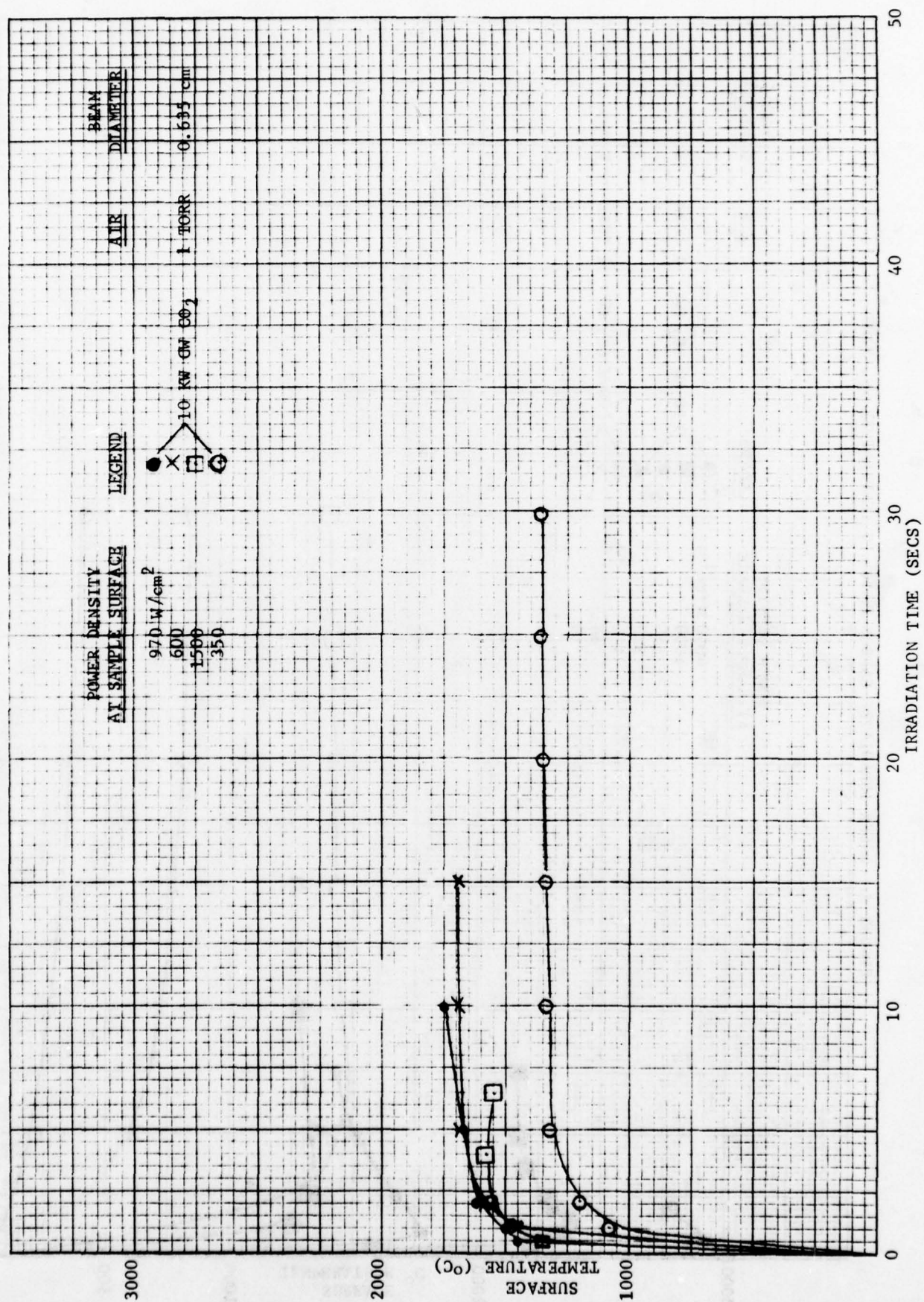


FIGURE 5. LASER IRRADIATED SILICA PHENOLIC TEMPERATURE VS. TIME

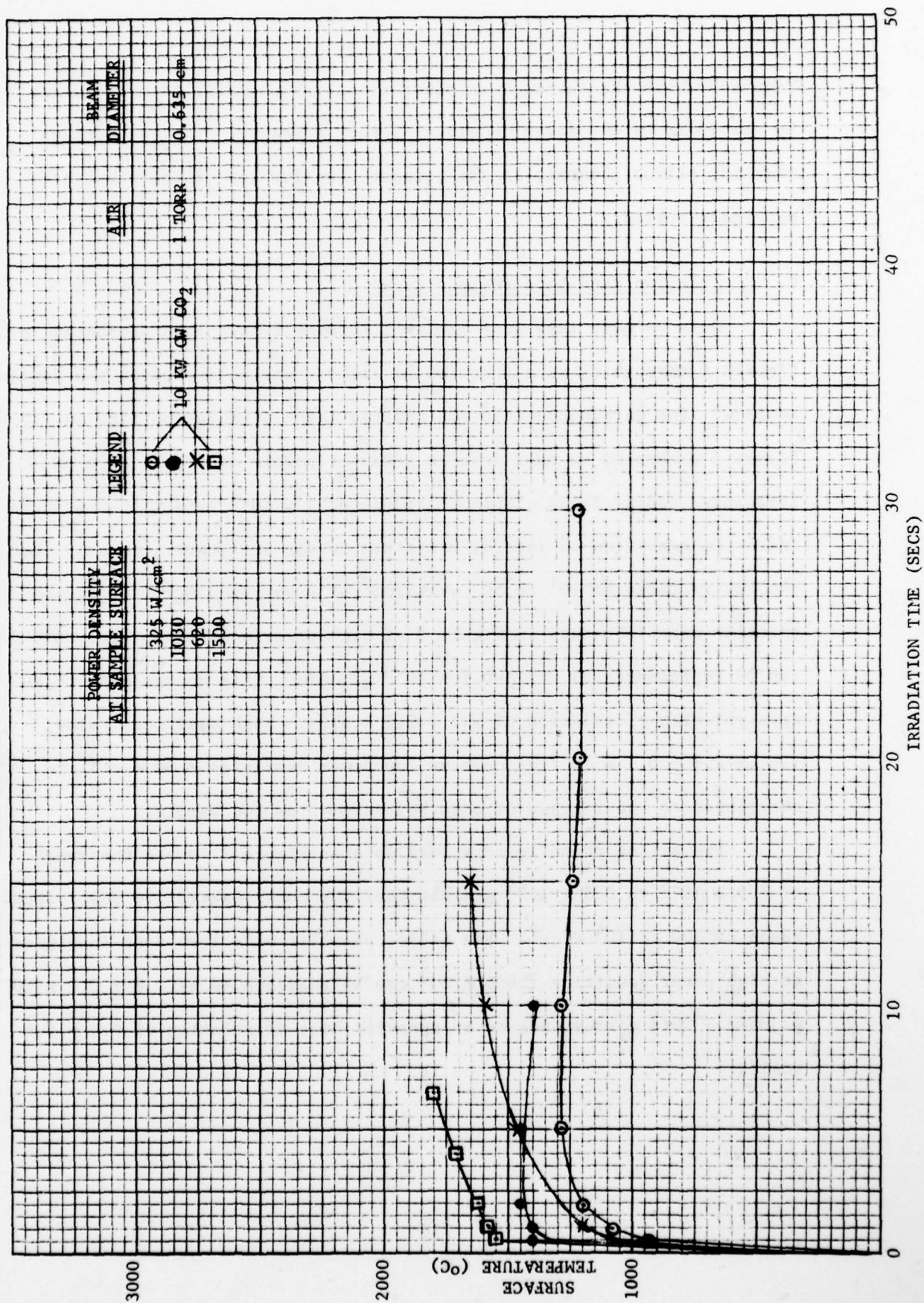
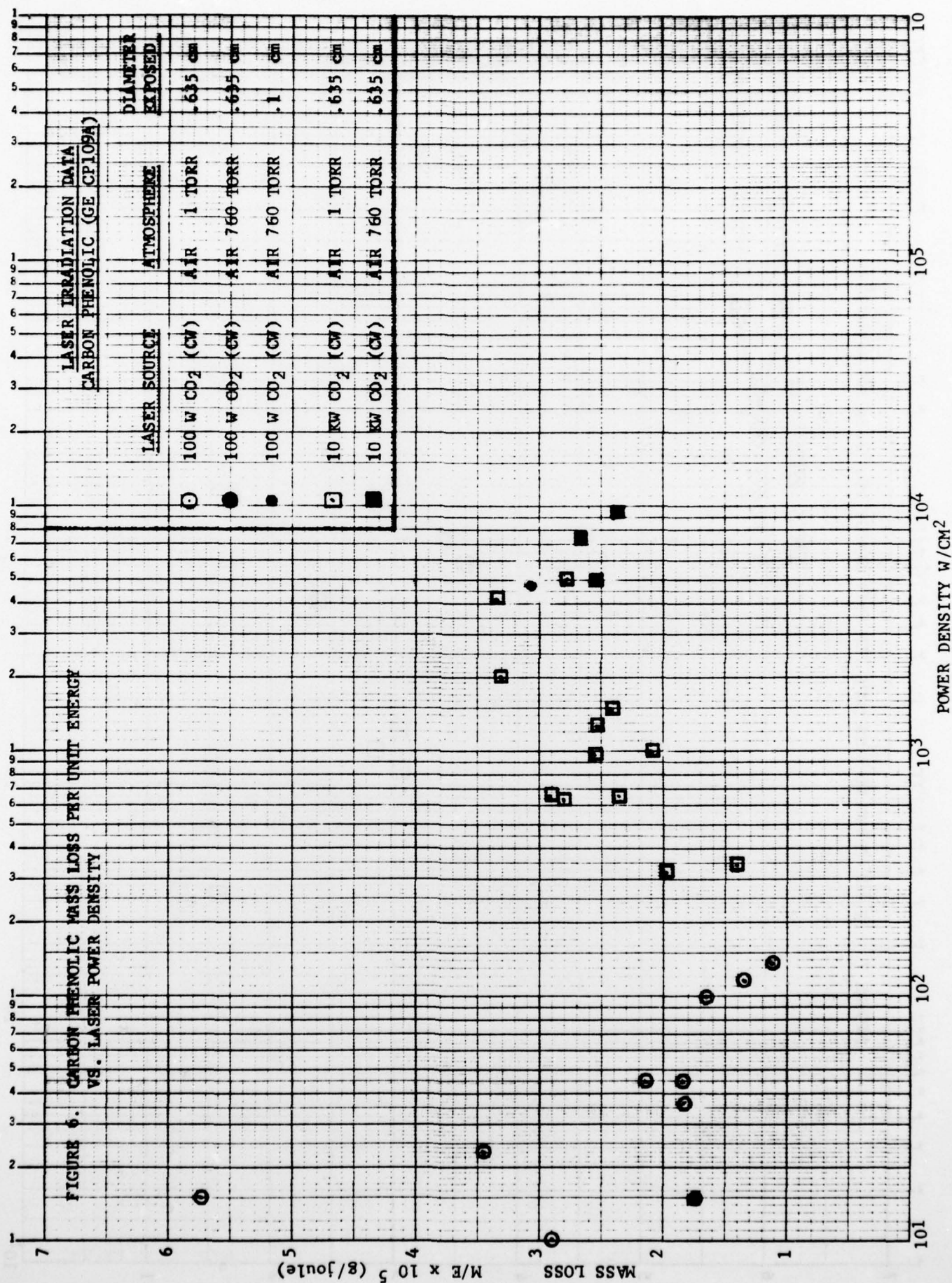


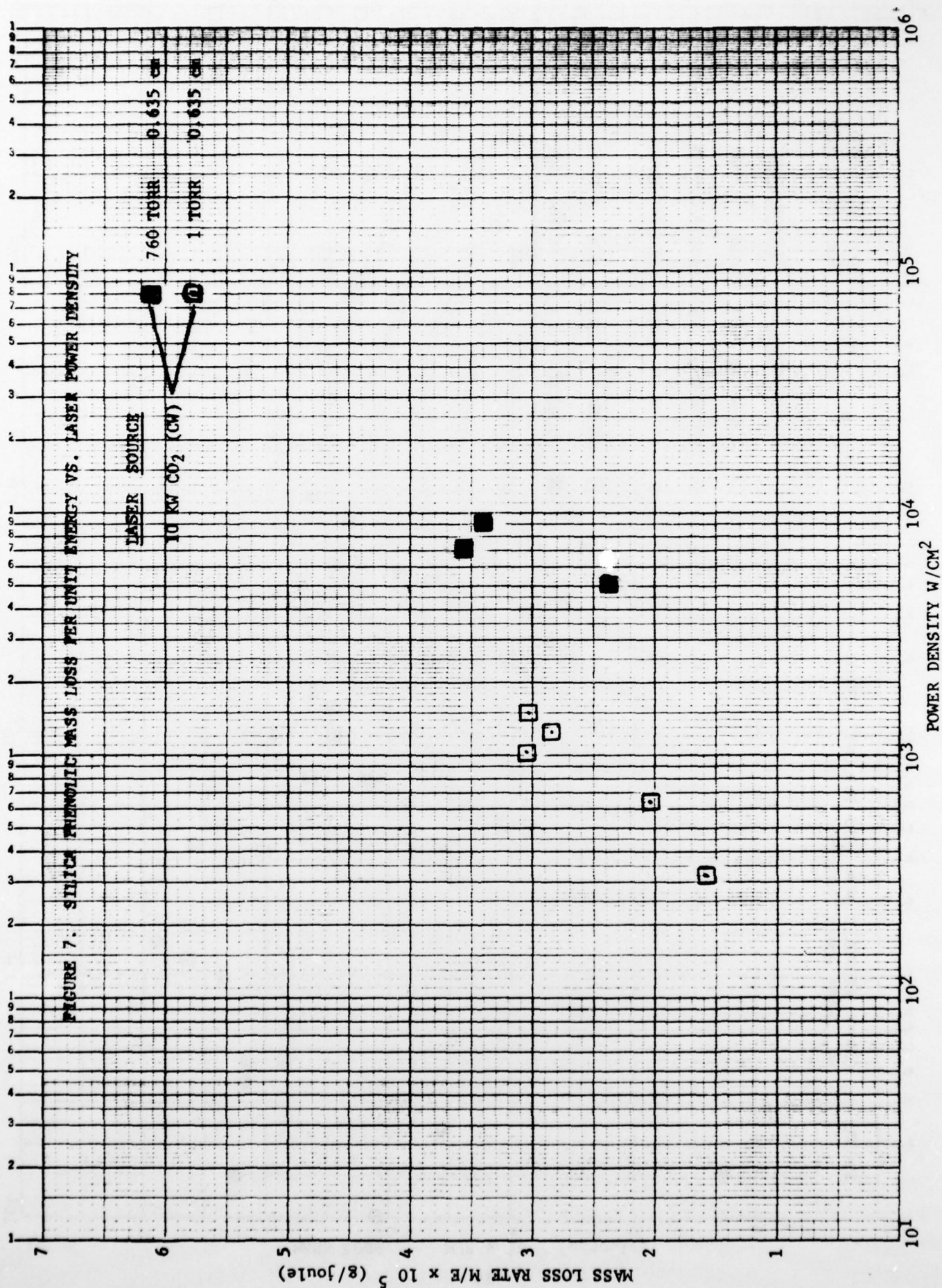
Figure 5 shows the temporal response of silica phenolic to irradiation by the 10 KW CW CO₂ laser in air at 1 torr pressure. The temperature rise for silica phenolic is far steeper than for any of the other R/V materials tested.

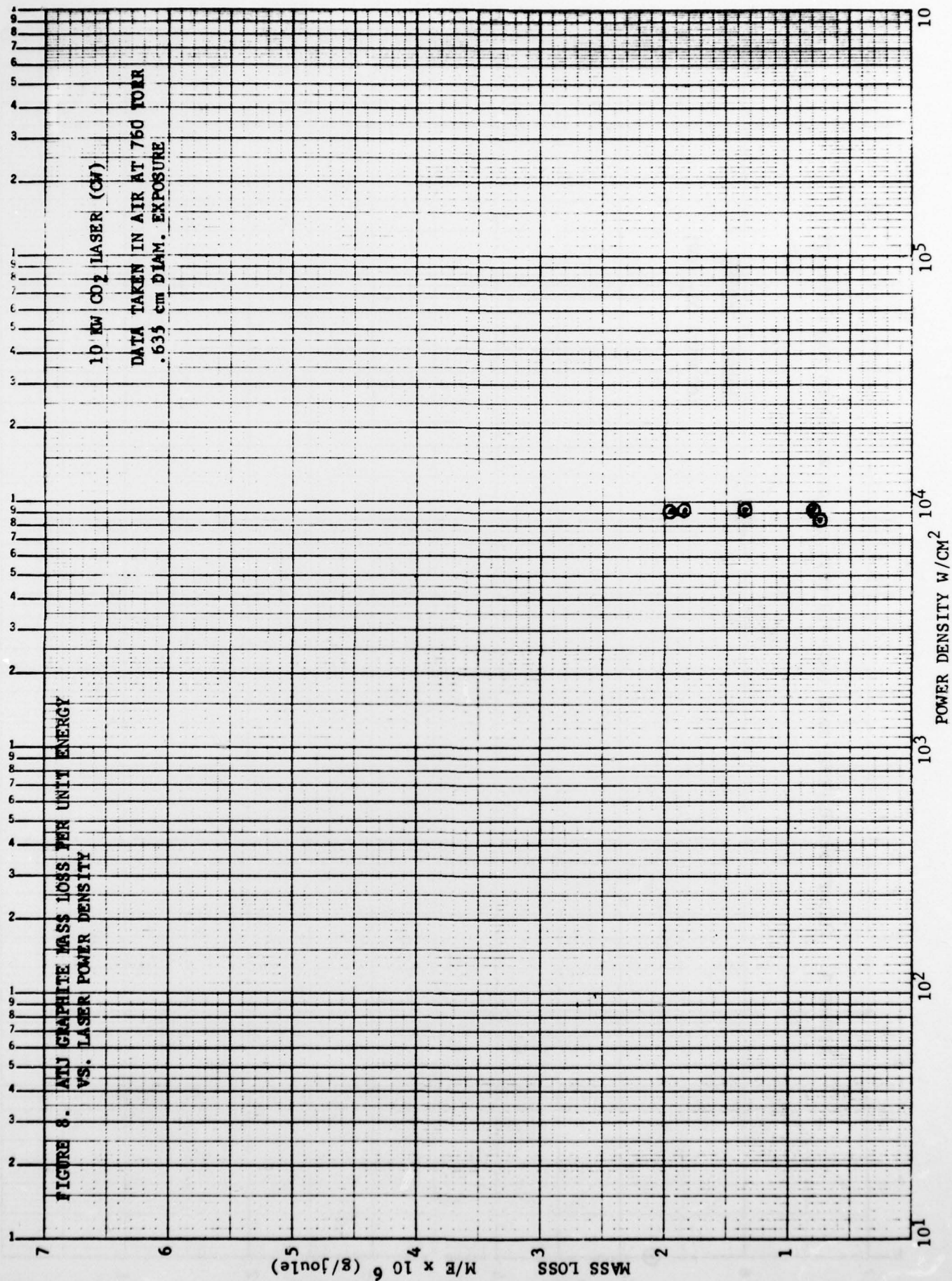
C. Ablation Rate Data (Weight Loss)

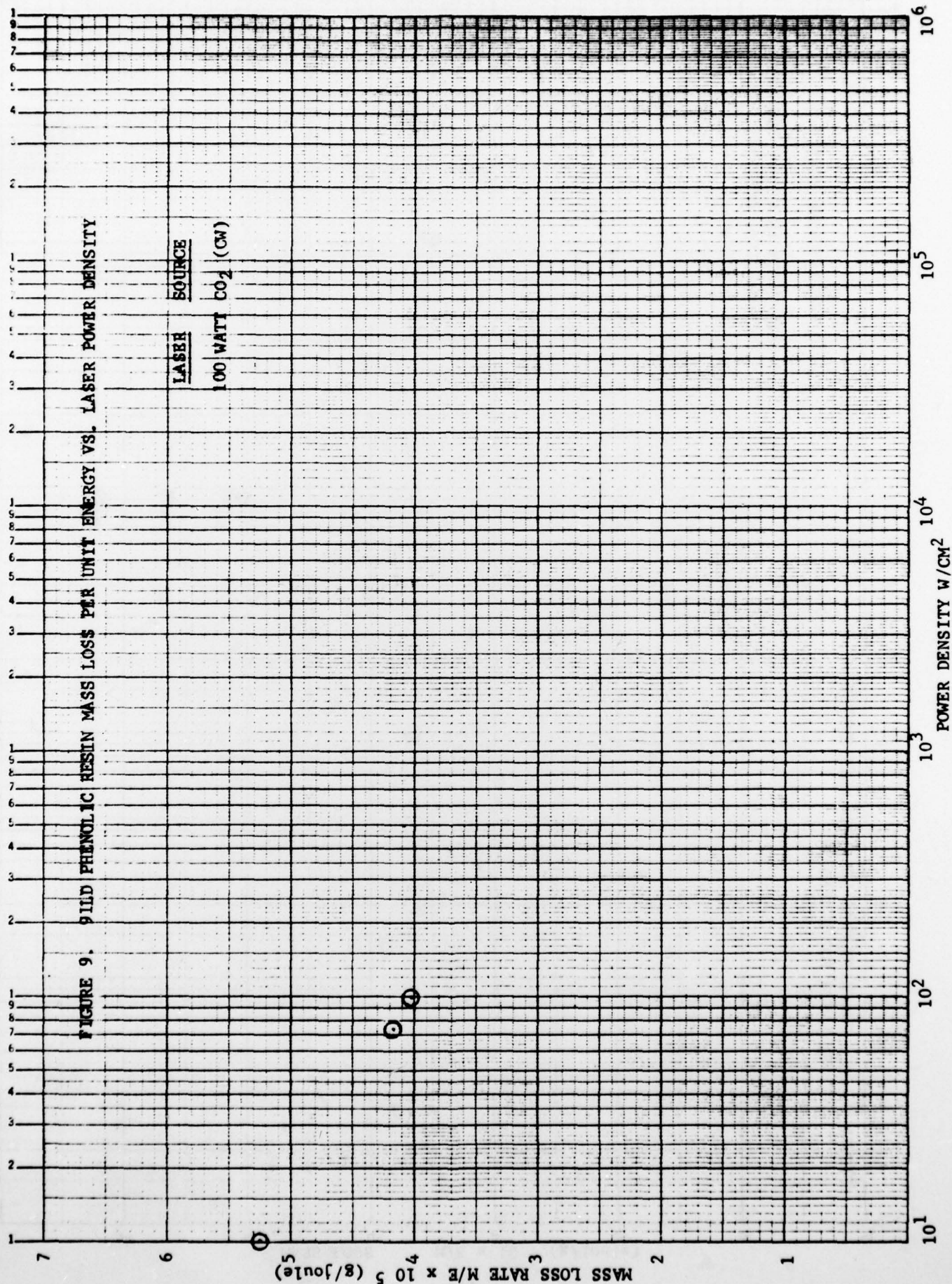
Data on mass loss from Tables 1 through 6 have been presented in Figures 6 through 14 as a function of laser power delivered at the sample surface. The carbon phenolic (GE CP109A) laser irradiation data in Figure 6 presented as mass loss in grams of sample lost per joule of irradiated energy falls within the range of 1×10^{-5} to 3×10^{-5} g/joule (with the exception of several tests run in air at 1 torr pressure) over the entire range of 10^1 to 10^4 watts/cm² of power density. The carbon phenolic mass loss data in Figure 6 at high power densities, i.e., 600 W/cm² can be represented as $2.7 \pm 0.7 \times 10^{-5}$ g/joule. This corresponds to an apparent heat of ablation of 9.5 ± 2.5 kcal/g. High mass loss rates, i.e., $\geq 2.7 \pm 0.7 \times 10^{-5}$ g/joule at low power densities < 30 W/cm² are attributed to the release of adsorbed water from the samples.

The silica phenolic mass loss data in Figure 7 covers the range of 1.5×10^{-5} to 3.5×10^{-5} g/joule over the more restrictive power density range of 300 to 10^4 W/cm². The silica phenolic mass loss data in Figure 7 at high power densities ≥ 600 W/cm² can be represented as $2.8 \pm 0.8 \times 10^{-5}$ g/joule. This corresponds to an apparent heat of ablation of 9.3 ± 2.6 kcal/g.









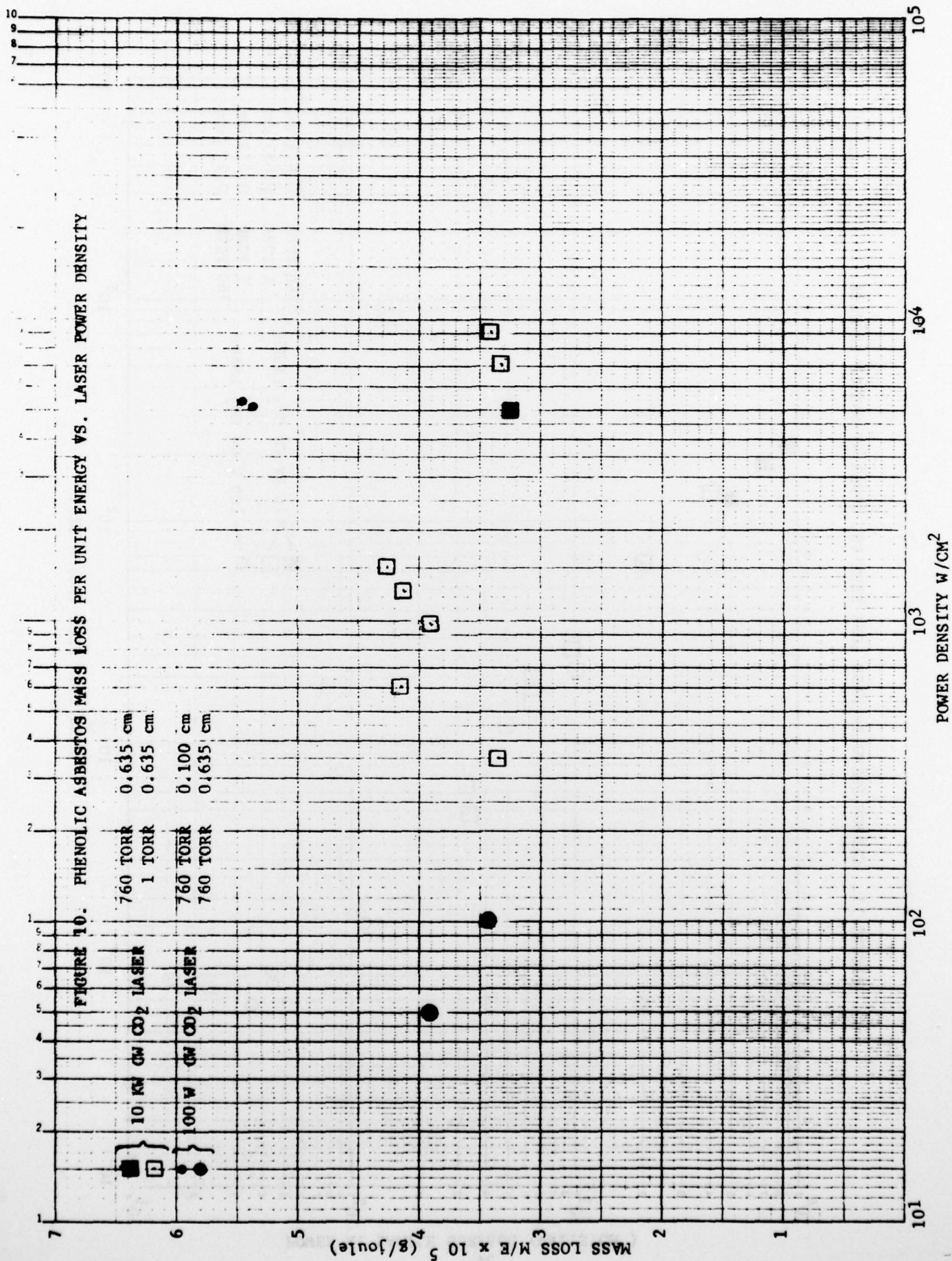


FIGURE 11. CARBON PHENOLIC MASS LOSS FLUX VS. POWER AT SAMPLE SURFACE

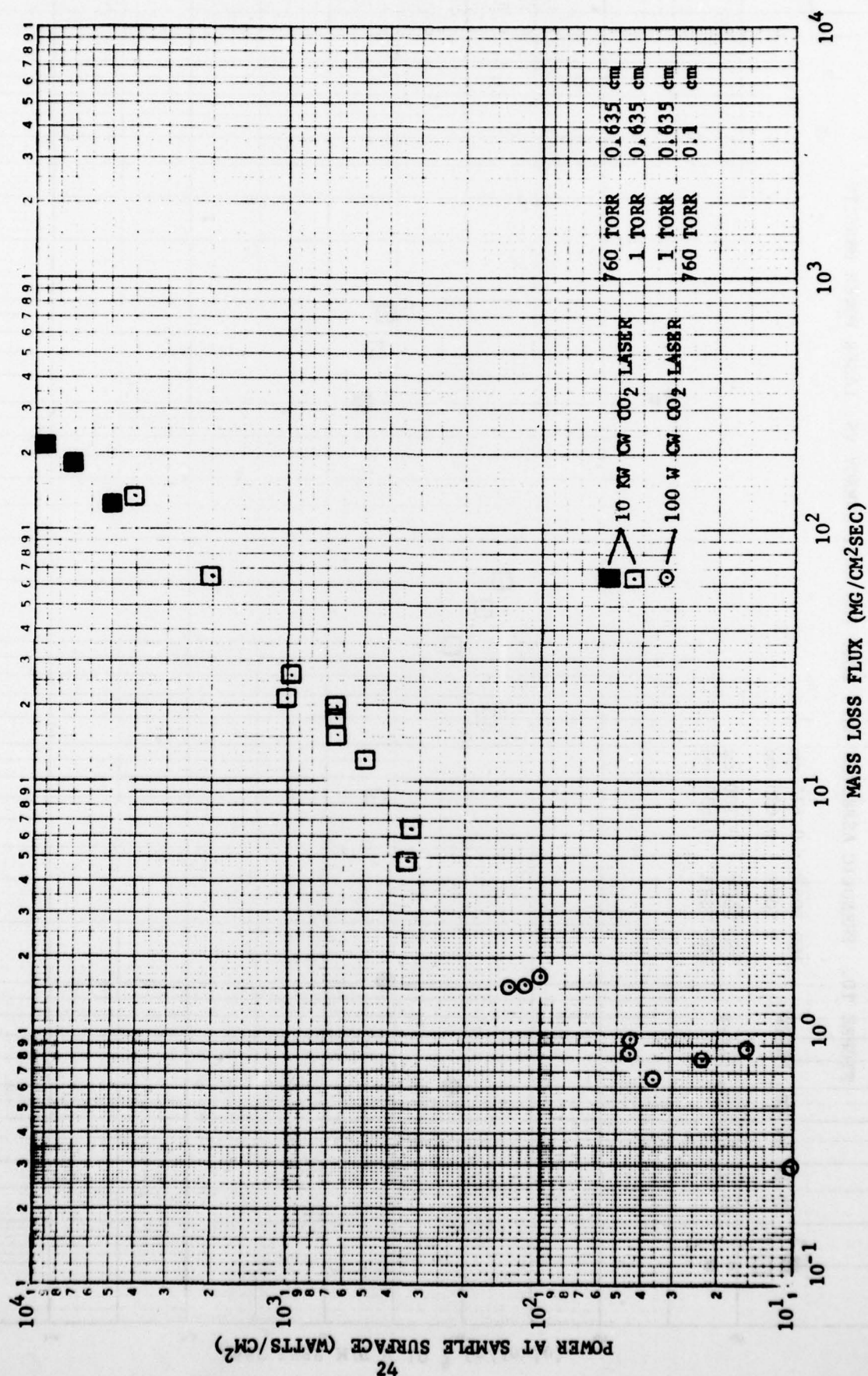


FIGURE 12. SILICA PHENOLIC MASS LOSS FLUX VS. POWER AT SAMPLE SURFACE

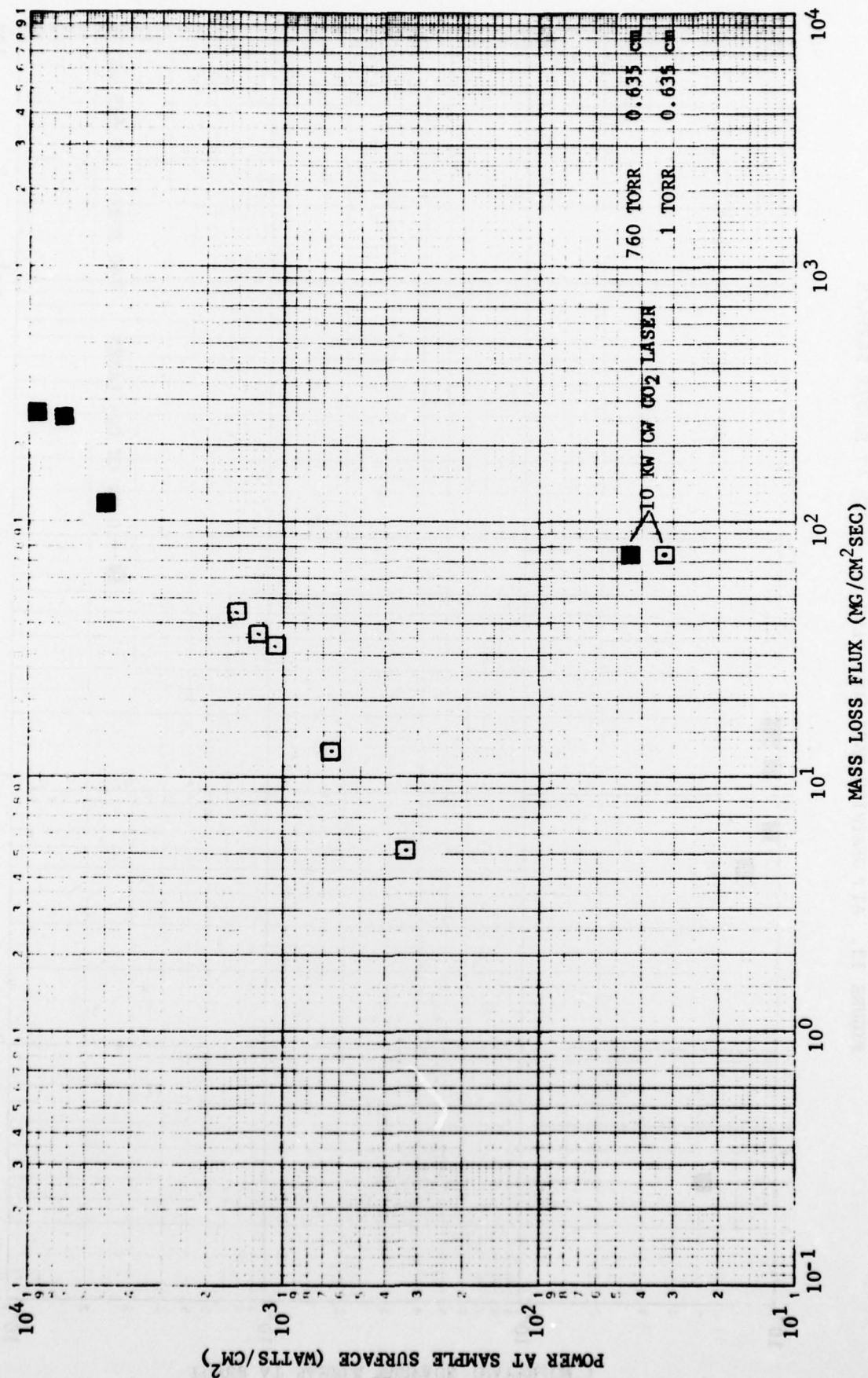


FIGURE 13. ATJ GRAPHITE MASS LOSS FLUX VS. POWER AT SAMPLE SURFACE

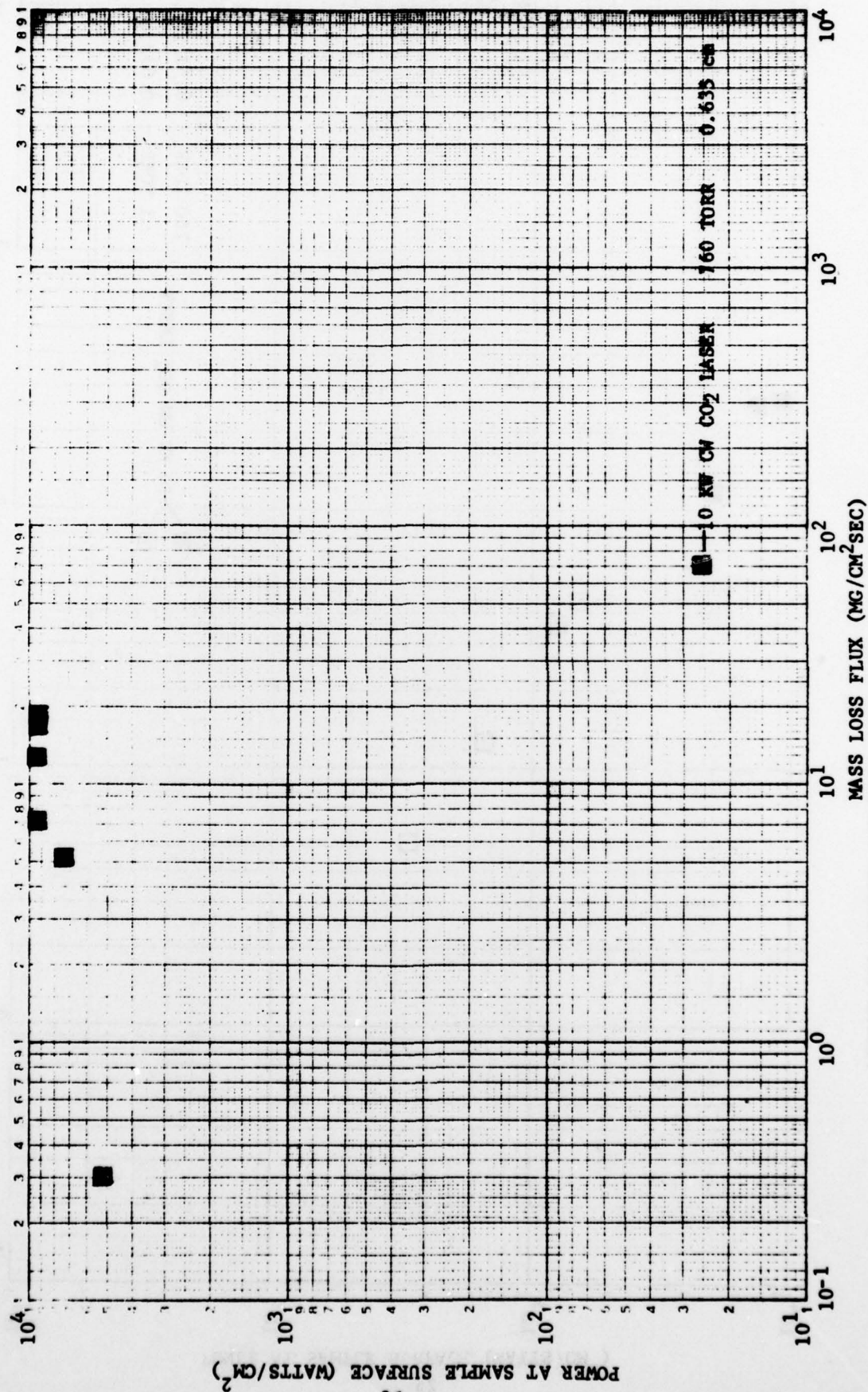
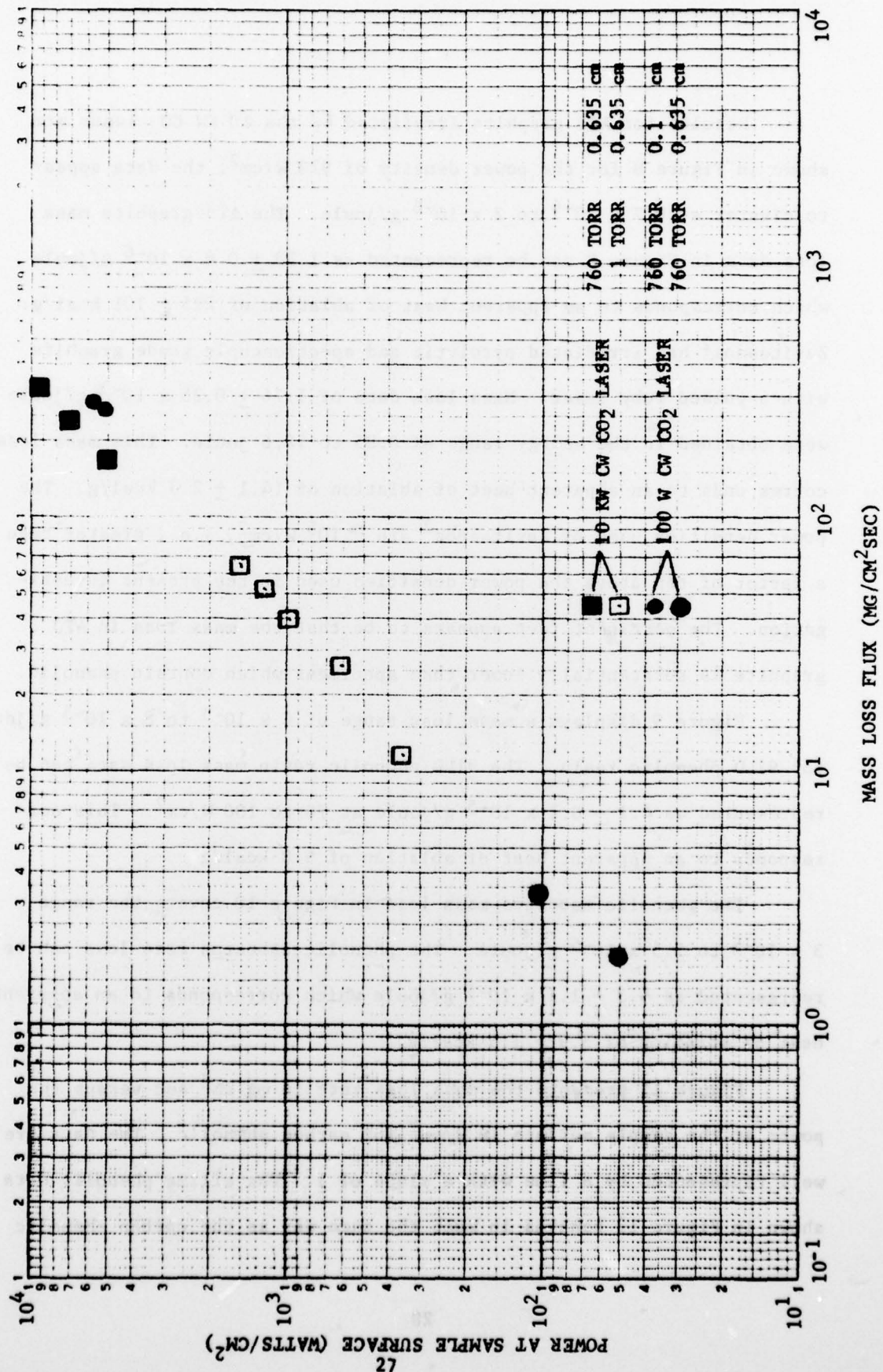


FIGURE 14. PHENOLIC ASBESTOS MASS LOSS FLUX VS. POWER AT SAMPLE SURFACE



Results for ATJ graphite irradiated by the 10 KW CO₂ laser are shown in Figure 8 for the power density of 920 W/cm²; the data appear to cluster at 0.7×10^{-6} to 2×10^{-6} g/joule. The ATJ graphite mass loss data in Figure 8 can be represented as $1.33 \pm 0.6 \times 10^{-6}$ g/joule which corresponds to an apparent heat of ablation of 225 ± 101 kcal/g. Zavitsanos² has irradiated pyrolytic and spectroscopic grade graphite with a pulsed ruby laser. Mass loss data of $1.76 \pm 0.35 \times 10^{-5}$ g/joule were obtained in the energy range of 0.85 to 12.6 joule. This mass loss corresponds to an apparent heat of ablation of 14.1 ± 2.8 kcal/g. The power densities used by Zavitsanos² are $\geq 10^5$ W/cm², i.e., greater than a factor of ten above the power densities used in the present investigation. The pertinent fact appears to be that the mass loss in ATJ graphite is substantially lower than specimens which contain phenolic.

Figure 9 displays a mass loss range of 4×10^{-5} to 8×10^{-5} g/joule for 91LD phenolic resin. The 91LD phenolic resin mass loss data can be represented as $4.1 \pm 0.1 \times 10^{-5}$ g/joule at 70 to 100 W/cm². This corresponds to an apparent heat of ablation of 5.8 kcal/g.

The phenolic asbestos mass loss in Figure 10 covers the range 3×10^{-5} to 5.5×10^{-5} g/joule. The phenolic asbestos mass loss can be represented as $4.3 \pm 1.1 \times 10^{-5}$ g/joule which corresponds to an apparent heat of ablation of 5.9 ± 1.5 kcal/g.

Figure 11 presents the mass loss rate in mg/cm² sec versus the power at the sample surface in W/cm² for carbon phenolic. The data are well represented by a line with a slope of 1. The silica phenolic data shown in Figure 12 behaves in much the same way as the carbon phenolic

data cited above. The ATJ mass flux data in Figure 13 demonstrates the low volatility of this specimen relative to phenolic-containing materials upon irradiation by laser. The phenolic asbestos mass flux loss data in Figure 14 exhibits a slope of one when plotted versus power density in the same manner as other phenolic-containing specimens.

D. Gaseous Species

A series of experiments was conducted in which the gaseous ablation products were sampled during irradiation using a Bendix time-of-flight Mass Spectrometer. This work was done with the pyrex vacuum system described in Figure 1. A specially designed gas sampling valve which allows direct sampling into the mass spectrometer at pressures up to one atmosphere was used in these experiments. The material examined was 91LD phenolic resin. The power range was 10 to 100 W/cm² and the exposure time was 60 seconds.

Preliminary analysis of the mass spectra indicates the following: NH₃ and CH₄ at 10 W/cm² power; from 25 to 100 W/cm² NH₃, CH₄, 2-propanol, 2-butanol, C₂H₆, C₃H₈, C₄H₁₀ and C₅H₁₂. These results are displayed in Table 7.

TABLE 7. MASS SPECTRA OBSERVED DURING LASER HEATING OF PHENOLIC RESIN (GE 9ILD)

LE#	LASER POWER W/CM ²	TIME AT WHICH MASS PEAK APPEARED (SEC.)	MASS PEAK	POSSIBLE SPECIES
10	10	30 45 60	12 → 17	NH ₃ , CH ₄
	23 73 87 102	(END OF RUN) X — X	12 → 17	
12	23 73 87 102	X	30, 31, 45, 59	[2-PROPANOL 2-BUTANOL]
11	23 73 87 102	X — X	30, 31, 45, 59	
14 11 13	23 73 87 102	X — X	27, 29, 91, 92	[C ₂ H ₆ C ₃ H ₈ C ₄ H ₁₀ C ₅ H ₁₂]

IV. REFERENCES

1. Smith, D.M., Thibault, R.J. and Horne, T.T., "10 KW CO₂ Laser Test Facility for Vulnerability and Hardening Programs", presented at 3rd DoD Conference on Laser Effects, Vulnerability and Counter-measures (1977).
2. Zavitsanos, P.D., "Mass Spectrometric Analysis of Carbon Species Generated by Laser Evaporation", Carbon 1968, Vol. 6, pp. 731-737, Pergamon Press, NY.